

# X-ray interferometry of surfaces with Fresnel mirrors

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With a small double-mirror setup, we used grazing-x-ray interferometry to study nanometric steps. These one-dimensional steps were microfabricated upon the surface of one of the two mirrors; the other mirror provided the reference wave. Two geometries were studied. In the longitudinal case in which the x rays are parallel to the step edges, it is straightforward to determine the step size. In the transverse case, one deals with Fourier holography, and a reconstruction process for a phase object had been demonstrated for the case of a single step. © 2000 Optical Society of America

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## 1. Introduction

Until a few years ago, synchrotron radiation sources had appreciable spatial coherence only for soft x rays, and interferometric methods (e.g., determination of optical constants of materials<sup>1</sup>) had been developed in the 2–10-nm range. Recently sources such as that at the European Synchrotron Radiation Facility in Grenoble, France, attained sufficiently large spatial coherence in the hard-x-ray range to allow experiments on Fresnel-zone phase contrast imaging<sup>2–4</sup> and photon correlation (speckle) scattering<sup>5–7</sup> to be performed. The spatial coherence of an optical wave front, defined quantitatively by the mutual intensity function, can be experimentally determined—and is in fact operationally defined—by Young's experiment with a variable slit separation.

For an x-ray beam it might be more convenient to use reflecting optics because it is not easy to achieve thick double slits with a short and variable distance between them and because the edges of the slits reflect and add spurious radiation to the interference pattern. Moreover, a mirror setup has more degrees of freedom, and we have shown that with two small Fresnel mirrors at grazing incidence it is possible to measure the spatial coherence in both the vertical

and the horizontal directions.<sup>8</sup> Here we analyze the possibility of using this two-mirror apparatus to make holograms of nanometric surface reliefs upon a flat surface.

## 2. Two-Mirror Setup

The geometry of the experiment is shown in Fig. 1. A synchrotron beam is reflected by two mirrors at a grazing angle of incidence  $\theta$ . The mirrors have size  $A$ , separation distance  $D$ , and relative height  $h$ ;  $L$  is the distance from the detector to the mirrors. Typical values are  $D$ , 1 cm;  $A$ , 1 mm;  $h$ , 0–10  $\mu\text{m}$ ; and  $L$ , and 5 m. The angle of incidence is less than 1 mrad, and the reflected beam at the detector is  $z \sim 10$  mm above the direct beam. By changing the angle of incidence we change the relative distance between the mirrors across the beam. We can also rotate one of the mirrors with respect to the other. Rotation about the direction of propagation the horizontal coherence properties to be studied, whereas rotation about the other horizontal axis displaces the reflected beam in the vertical plane. Elevating one of the mirrors produces a variation of the distance between the mirrors as seen from the detector, and the opposite variation as seen from the source (Figs. 1 and 2).

## 3. Longitudinal Geometry

Vertical displacement of one mirror by  $h$  produces a variation of the optical path length of the reflected beam  $\Delta\varphi = 4\pi h/\lambda \sin \theta$ . At 0.1-nm wavelength and 1-mrad angle of incidence,  $h = 50$  nm produces a  $2\pi$  phase shift. Special care has to be taken for stability. We used this effect to study model systems, i.e., mirrors consisting of steps a few hundred nanometers in size produced by either lithography or UHV deposition [Fig. 3(a)]. If the transverse width of the steps is a few hundred micrometers wide, we can consider

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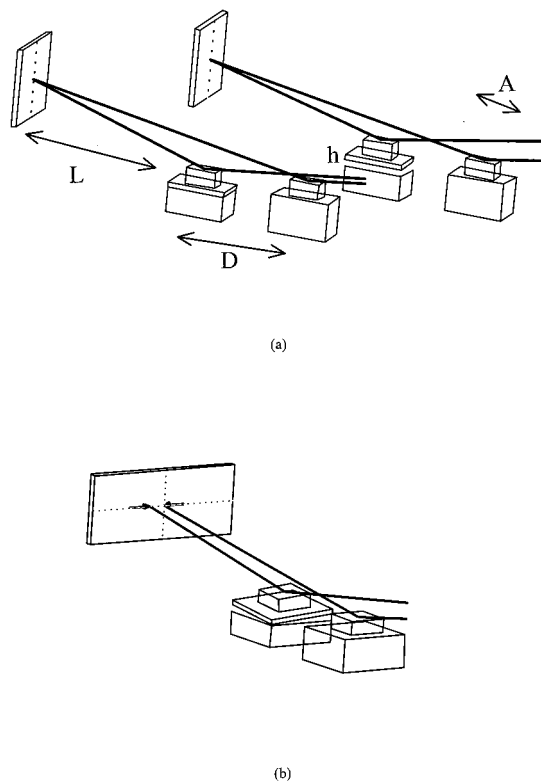


Fig. 1. (a) Experimental setup. At the right one of the mirrors is elevated an amount  $h$ . (b) Horizontal tilt.

the detector to be in the near field, and the path-length difference produces a phase shift in the interference pattern. Figure 3(b) shows typical steps measured on a molecular beam epitaxy sample [Fig. 3(a)] at a 0.44-mrad grazing angle. Agreement of the experimentally determined step size was found within the specifications of the mirror fabrication.

#### 4. Transverse Geometry

We considered the case of a simple step (two terraces) with a step height of 10 nm. The terrace widths in this case are compressed because of the grazing incidence. The detector can be considered to be in the far field; i.e., we are in the Fourier holography regime.

##### A. Measurement

The first aspect to consider is the inclination of the mirrors. Mirrors of size  $A$  and distance  $D$  are seen from the detector with projected cross section size  $a = A \sin \theta$  and distance  $d = D \sin \theta$ , where  $\theta \sim z/L$  is the elevation angle of the detector. The interference pattern produced by two rectangular slits has the well-known expression  $I(z) \propto \text{sinc}[kz a(z)/L] \sin[kz d(z)/L + \varphi]$ . This shows that the spatial frequency of oscillation is linearly changing with  $z$ , as can be seen from the spectrogram. The measured intensity as a function of CCD pixel height ( $z$ ) is shown in Fig. 4(a).

We obtain the spectrogram [Fig. 4(b)] by plotting the Fourier transforms of sections of the image. Fig-

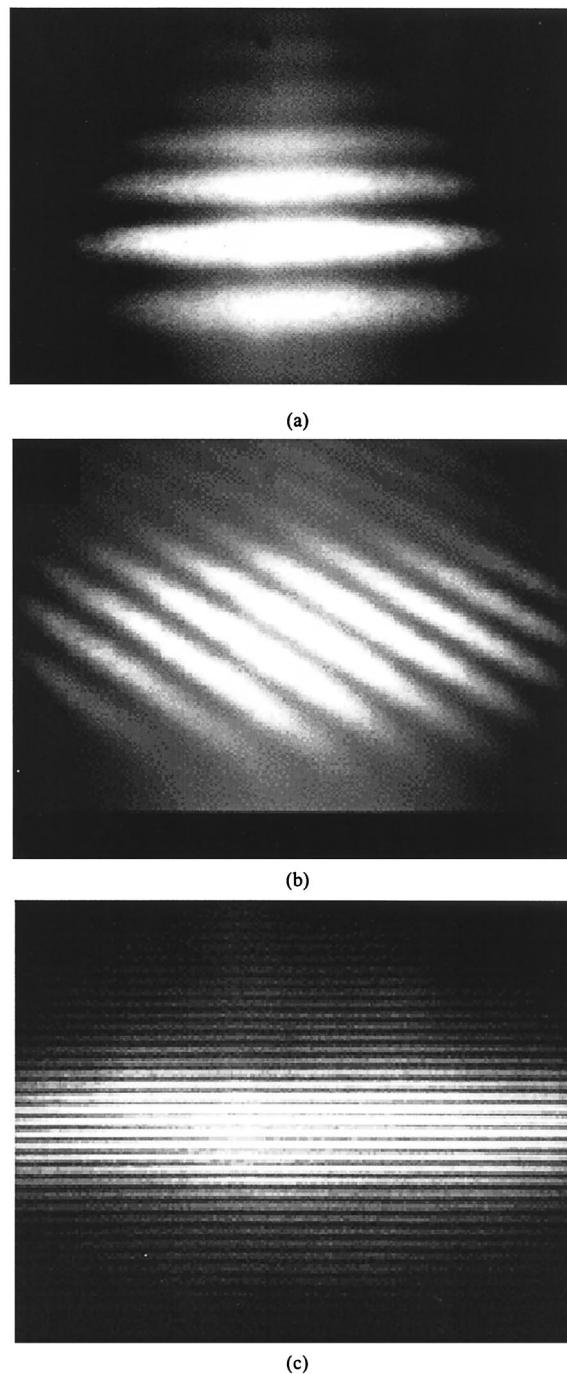


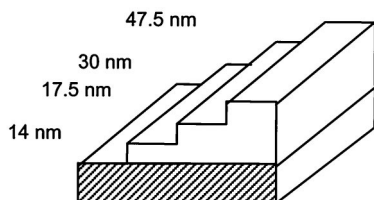
Fig. 2. Interference pattern produced by the two mirrors at 10.4 keV recorded by a CCD: (a) parallel mirrors, (b) tilt of one mirror, (c) relative distance from the detector decreased by elevation of one mirror.

ure 5 shows the intensity (and its spectrogram) of the hologram corrected for the inclination effect.

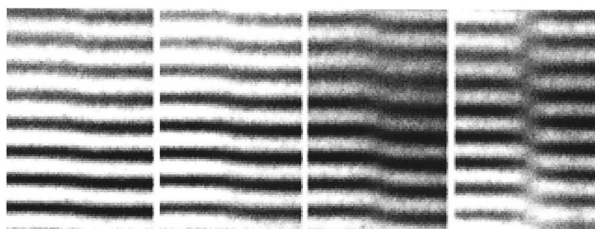
The intensity produced by the two mirrors is

$$I(z) = I_r + I_o + 2\sqrt{I_r I_o} \sin[k\varphi z + \delta\varphi(z)],$$

where  $I_r$  and  $I_o$  are the intensities produced by the reference mirror and the object mirror, respectively, and  $\varphi = a/L$  is the angular distance between the two



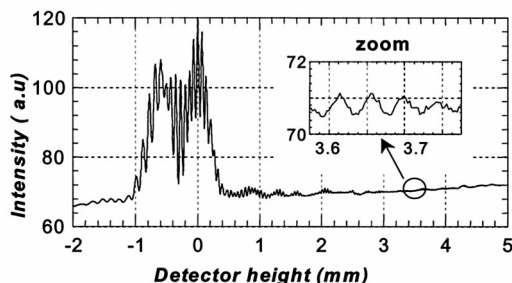
(a)



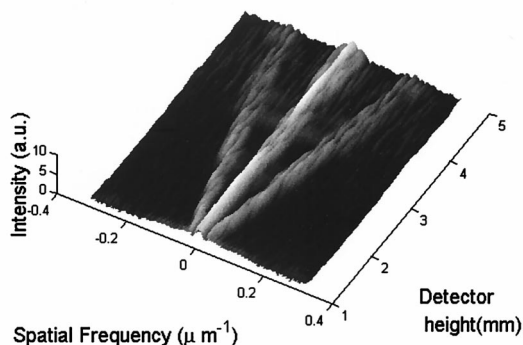
(b)

Fig. 3. (a) Stepped mirror used for the experiment. Step heights are indicated. (b) Hologram of the stepped mirror in longitudinal geometry at 10.4 keV,  $\theta = 0.44$  mrad.

mirrors as seen from the detector. The phase difference  $\delta\phi$  between the complex amplitude of the object and reference waves is assumed to be slowly changing with  $z$ .

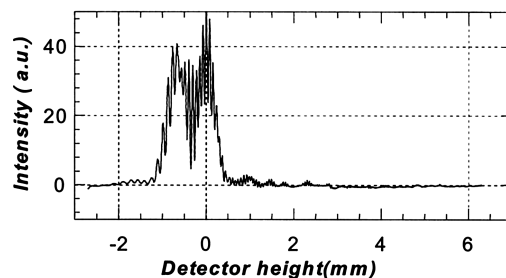


(a)

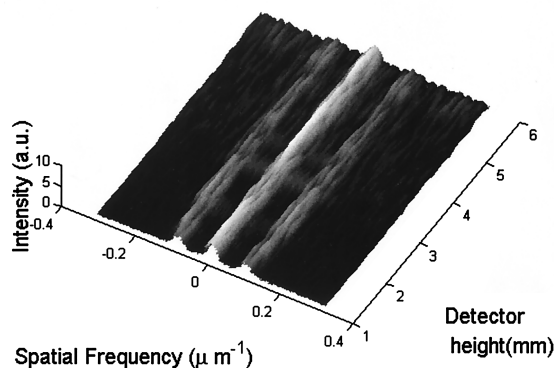


(b)

Fig. 4. (a) Hologram of the mirror in transverse geometry ( $E = 10.4$  keV,  $\theta = 0.8$  mrad,  $D = 20$  mm). (b) Spectrogram. The original signal in (a) is divided into parts, and each part is Fourier transformed. The picture shows the signal in phase space of frequency space.



(a)



(b)

Fig. 5. (a) Hologram and (b) spectrogram corrected for the inclination effect  $\{I'(z) = I[z(\theta + z/L)]\}$ .

## B. Principles of Object Reconstruction

By Fourier filtering we separate the low-frequency term  $I_l(z) = I_r + I_o$  and the oscillating (high-frequency) term  $I_h(z) = \sqrt{I_r I_o}$ . It is easy to invert the system of equations:

$$I_{r,o} = \frac{1}{2} [I_l \pm (I_l^2 - 4I_l I_o)^{1/2}]. \quad (1)$$

We obtain two solutions; if the two beams are intentionally not well superposed at the detector we know which one is higher and thus can disentangle them (Fig. 6).

Once the two intensities are separated we can use them to reconstruct the step. If we neglect the relative curvature of the wave fronts emerging from the

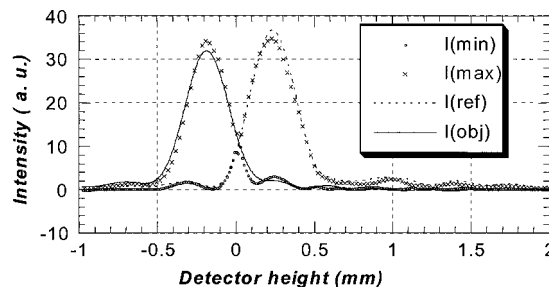


Fig. 6. Intensities of the two beams separated by application of Eq. (1) to the data.

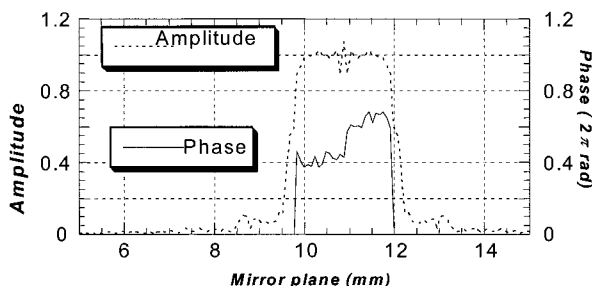


Fig. 7. Reconstruction of the stepped mirror. A phase step corresponds to the phase shift produced by the step.

two mirrors, the phase of the reference beam is 0 or  $\pi$ . Knowing the amplitude and the phase of the object wave, we can apply a Fourier transform to reconstruct the object mirror (Fig. 7).

The resolution is limited by the angular range of detection; the angular range in which the reference beam is not negligible is inversely proportional to the projected size of the reference mirror. Thus the object features that can be studied are in the range of the size of the reference mirror. For a mirror of few millimeters, at 1-mrad incidence, the reference beam is few micrometers. In any case, for the object (the step) to be observable it must produce a nonnegligible phase shift ( $2h \sin \theta / \lambda > 0.1$ ); hence a step size as small as  $\sim 5$  nm can be detected.

## 5. Conclusions

Using an apparatus developed for measurement of the spatial coherence of a hard-x-ray beam, we have analyzed the possibility of getting holographic information on nanometric reliefs upon a flat surface. We have measured stepped surfaces in both the transverse and in the longitudinal directions.

This study has shown that reconstruction of the object mirror is possible, although with a resolution not comparable with that from an atomic-force microscope. However, this apparatus could be used for the study of time-varying surface reliefs under external constraints (e.g., a magnetic field), and one could

also make it element selective by tuning the x-ray wavelength. Therefore it can be interesting to try experiments in which these specific advantages can be utilized.

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## References

1. F. Polack, D. Joyeux, J. Svatos, and D. Phalippou, "Applications of wavefront division interferometers in soft x rays," *Rev. Sci. Instrum.* **66**, 2180–2183 (1995).
2. A. Snigirev, I. Snigireva, V. Kohn, and S. Kuznetsov, "On the possibilities of phase-contrast micro-imaging by coherent high energy synchrotron radiation," *Rev. Sci. Instrum.* **66**, 5486–5492 (1995).
3. I. Schelokov, O. Hignette, C. Raven, A. Snigirev, I. Snigireva, and A. Souvorov, "X-ray interferometry technique for mirror and multilayer characterization," in *Multilayer and Grazing Incidence X-Ray/EUV Optics III*, R. B. Hoover and A. B. Walker, eds., *Proc. SPIE* **2805**, 282–291 (1996).
4. P. Cloetens, J. P. Guigay, C. DeMartino, and J. Baruchel, "Fractional Talbot imaging of phase gratings with hard x rays," *Opt. Lett.* **22**, 1059–1061 (1997).
5. S. Brauer, G. B. Stephenson, M. Sutton, R. Brünig, E. Dufresne, S. G. J. Mochrie, G. Grübel, J. Als-Nielsen, and D. L. Abernathy, "X-ray intensity fluctuation spectroscopy observations of critical dynamics in  $\text{Fe}_3\text{Al}$ ," *Phys. Rev. Lett.* **74**, 2010–2013 (1995).
6. E. Gluskin, I. McNulty, P. J. Viccaro, and M. R. Howells, "X-ray intensity interferometry of surfaces with Fresnel mirrors," *Nucl. Instrum. Methods A* **319**, 213–218 (1992).
7. Y. Kunimune, Y. Yoda, K. Izumi, M. Yabashi, X.-W. Zhang, T. Harami, M. Ando, and S. Kikuta, "Two-photon correlations in x-rays from a synchrotron radiation source," *J. Synchrotron Radiat.* **4**, 199–203 (1997).
8. S. Marchesini and R. Coisson, "Two-dimensional coherence measurements with Fresnel mirrors," *Opt. Eng.* **53**, 3597–3601 (1996).
9. K. Fezzaa, F. Comin, S. Marchesini, R. Coisson, and M. Belakhovsky, "X-ray interferometry at ESRF using two coherent beams from Fresnel mirrors," *J. X-Ray Sci. Technol.* **7**, 12–23 (1997).